1,012,495

PATENT SPECIFICATION

NO DRAWINGS

1,012,495

Date of Application and filing Complete Specification: Feb. 27, 1964.

No. 8286/64.

Application made in Japan (No. 8620) on Feb. 27, 1963.

Complete Specification Published: Dec. 8, 1965.

Ind x at acceptance:—C3 H1; G2 C(C9C, C9H1A, C9H3A2)

Int. Cl.:—G 03 c

COMPLETE SPECIFICATION

Improvements in or relating to Gelatin-Containing Compositions

We, FUJI SHASHIN FILM KABUSHIKI KAISHA, a corporation organized under the laws of Japan, of 210, Nakanuma, Minamiashigara-machi, Ashigarakami-gun, Kanagawa-ken, Japan, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to gelatin-containing compositions and is concerned, more particularly but not exclusively, with gelatin-containing compositions for use in the production of photo- or radio-sensitive materials.

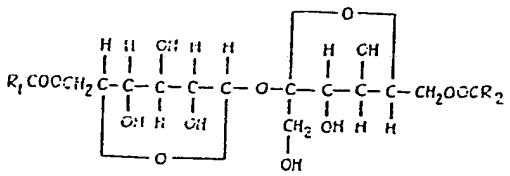
In the application of gelatin coatings to surfaces, particularly in the photographic industry, it is generally desired to apply one or more layers of a gelatin-containing composition to such surfaces uniformly and at good production speeds. In many cases, it is desired to apply a coating of a gelatin-containing composition over a previous coating, which may be based either on gelatin or on some other material, such previous layers being wet or dry. In such circumstances, the addition of a coating aid, such as saponin, to the coating composition is made with a view to obtaining the application of a smooth, even coating. In order to prevent the formation of bubbles and foams in the gelatin coating there may be added to the composition a synthetic surface-active agent. Saponin, although quite useful as a coating aid in many respects, is nevertheless a naturally occurring substance and therefore may show decided variations in quality from batch to batch. Moreover, bubbles and foams are frequently formed in gelatin coatings obtained from compositions containing saponin. Accordingly, various kinds of surface-active agents have come to be employed, particularly in the photographic industry. It is often stated that such surface-active agents are used not only as coating aids, but also as antistatic agents, solubilizers for colour-forming compounds, wetting agents in the processing solutions, water-mark reducing agents or development accelerators and so on. However, many of these surface-active agents have such specific effects that they can give satisfactory results only when used in specific gelatin compositions. In other words, many of the hitherto known surface-active agents are limited in their range of use.

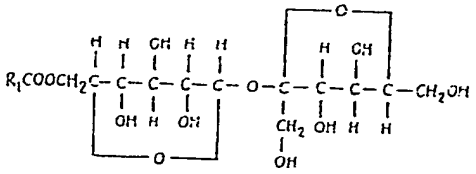
It is a particularly difficult problem to obtain a smooth coating from a composition consisting of colloidal silver suspended in a very dilute gelatin solution. Very often it is desired that the colloidal silver suspension be directly applied to a subbed film base to make an antihalation layer. In such a case, it is almost impossible to prepare a suspension of colloidal silver in aqueous gelatin or to coat it onto the subbed film in such a way that no bubbles or repellencies are produced.

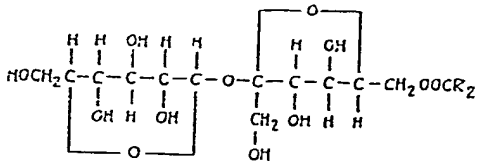
According to one aspect of the invention there is provided a composition comprising an aqueous dispersion of gelatin and, as a coating aid, a minor proportion of a sucrose mono- or di-ester of an aliphatic monocarboxylic acid, said acid having from 7 to 18 carbon atoms.

It has now been found that the coating properties of a gelatin-containing composition can be improved by adding to the composition a condensation product of sucrose with an aliphatic monocarboxylic acid. It is believed that the sucrose esters used in the present invention are represented by the following general formulae;

[*Price 4s. 6d.*]







wherein R_1 and R_2 are aliphatic hydrocarbon radicals having from 6 to 17 carbon atoms.

In the above general formulae, when the aliphatic hydrocarbon radical R_1 or R_2 contains less than 6 carbon atoms, the aliphatic sugar ester has little or no activity as a surface active agent, and when it contains more than 17 carbon atoms, the ester is hardly soluble in water. The compounds used in the present invention, are soluble in water and capable of forming an aqueous solution of more than 0.5% w/v concentration at 25° C. These compounds include, for example, sucrose monolaurate, sucrose monooleate, the sucrose monoesters of coconut oil aliphatic acids, sucrose dicaprylate, sucrose monomyristate and sucrose dicaprenate.

The sucrose esters, which are used in the present invention, can be prepared by dissolving sucrose in dimethylformamide, adding to the resulting solution an aliphatic ester of an aliphatic monocarboxylic acid, and heating the mixture on a hot water bath under a reduced pressure in the presence of an alkaline catalyst (see Industrial and Engineering Chemistry *48*, No. 9, pp. 1459—1462 (1956)).

The surface-active sucrose esters are advantageously added as coating aids to a gelatin-containing coating composition intended for photo- or radio-graphic use in amounts of from 0.05 g. to 100 g., and preferably from 1 g. to 50 g., per kg. of dried gelatin in the composition. The compounds may be used in admixture with one another or as a mixture with other surface-active agents.

The invention will be illustrated by the following Examples in which all percentages are on a w/v basis unless otherwise specified.

Example 1.

A silver chlorobromide photographic emulsion containing 7% gelatin and 5% silver halide in aqueous dispersion was prepared. There was then added thereto 0.2 g. of sucrose monolaurate per kg. of photographic emulsion. The resulting photographic emulsion was coated onto a paper base, which had previously been coated with baryta, and then cooled to gel without drying. An overcoating composition containing 0.2 g. of the sucrose monoesters of coconut oil aliphatic acids in 1 litre of a 2.5% aqueous gelatin solution, was coated on the chilled surface of the emulsion. After cool-setting, the resulting material was dried.

The use of the coating aid in the photographic emulsion layer and in the protective overcoat layer was successful; that is to say, the photographic emulsion layer and the protective coat layer were free from pin-holes, repellencies or other coating defects even when they were coated at a high speed.

EXAMPLE 2.

A run similar to that described in the preceding Example was carried out except that sucrose monooleate was used in the photographic emulsion as the coating aid instead of the monolaurate. Satisfactory results were obtained as in Example 1.

EXAMPLE 3.

A silver chlorobromide photographic emulsion containing 7% gelatin and 5% silver halide in aqueous dispersion was prepared. A 2.5% aqueous solution of sucrose monolaurate was added to quantities of the photographic emulsion in amounts ranging from 0.01 g. to 2.5 g. per kg. of said emulsion. The resulting photographic emulsions were then coated on samples of baryta paper, which were then dried.

It was found, that with an increase in the amount of sucrose monolaurate used, the numbers of repellency per m^2 of material were considerably reduced, as shown in Table I below.

TABLE I

Concentration of sucrose monolaurate in the emulsion (g/kg)	Number of repellency (number/m^2)
0.01	4
0.05	3
0.10	1
0.25	0
0.50	0
1.00	0
2.50	0

EXAMPLE 4.

A suspension of 2.5 g. of colloidal silver in 1 litre of a 6% aqueous gelatin solution was prepared. One gram of a 40% aqueous formaldehyde solution, as hardening agent, and 2 g. of the sucrose monoesters of coconut oil aliphatic acids were added thereto. The thus prepared dispersion was heated to a temperature of 40° C. and coated on to a cellulose triacetate base to a dry thickness of 2.0—2.5 microns. No bubbles were formed in the coating composition. The coating also had a very smooth appearance and was free of defects or repellencies. When a similar coating operation was carried out using 2 g. of saponin instead of the 2 g. of the sucrose monoesters of coconut oil aliphatic acids, it was quite difficult to avoid the formation of bubbles and foams.

EXAMPLE 5.

The coating operation described in Example 4 was repeated but 2 g. of sucrose dicaprylate were used in place of the 2 g. of the sucrose monoesters of coconut oil aliphatic acids. Excellent results, similar to those described in Example 4, were obtained.

EXAMPLE 6.

A high speed radiographic emulsion for medical X-ray film containing 7% gelatin and 6% silver iodobromide in aqueous dispersion was prepared. There was then incorporated in the photographic emulsion, 0.2 g. of sucrose monomyristate per kilogram of the emulsion. The resulting photographic emulsion was coated on to a cellulose triacetate base and cooled to gel without drying. A 2.5% aqueous gelatin solution which contained 0.1 g. of sodium long chain alkyl benzenesulphonate in 1 litre of gelatin solution was overcoated on the wet emulsion layer. After being cooled

to gel, these coatings were dried. In the coating operation, the ease of applying the overcoating to the wet photographic emulsion layer was superior to that observed when using saponin in the emulsion instead of the sucrose monomyristate. The coating also had a very smooth appearance and was free of defects, such as pinholes caused by bubbles in the coating composition. The antistatic properties of the film thus produced were also very good.

WHAT WE CLAIM IS:—

1. A composition comprising an aqueous dispersion of gelatin and, as a coating aid, a minor proportion of a sucrose mono- or di-ester of an aliphatic monocarboxylic acid, said acid having from 7 to 18 carbon atoms.

2. A composition, for use in the production of photo- or radio-graphic materials, comprising an aqueous dispersion of gelatin and from 0.05 to 100 g. of sucrose mono- or di-ester of an aliphatic monocarboxylic acid, said acid having from 7 to 18 carbon atoms, per 1 kg. of dried gelatin in the composition.

3. A gelatin-containing composition as claimed in Claim 1 or 2, wherein said aliphatic monocarboxylic acid is monolauric acid.

4. A gelatin-containing composition as claimed in Claim 2 or in Claims 2 and 3, wherein said composition further includes colloidal silver suspended therein.

5. A gelatin-containing composition as claimed in Claim 2 or in Claims 2 and 3, wherein said composition comprises a gelatin-silver halide photo- or radio-graphic emulsion.

6. A gelatin-containing composition as claimed in Claim 2, substantially as described in any one of the foregoing Examples 1 to 6.

7. A photo- or radio-graphic material comprising a coating formed from a gelatin-containing composition as claimed in Claim 2, 4, 5 or 6 or in Claim 3 when appendant to Claim 2.

8. In a process for the production of a photo- or radio-graphic material, the step of applying to a support a gelatin-containing composition as claimed in Claim 2, 4, 5 or 6, or in Claim 3 when appendant to Claim 2.

9. A photo- or radio-graphic material whenever prepared by the process claimed in Claim 7.

HASELTINE, LAKE & CO.,
Chartered Patent Agents,
28 Southampton Buildings, Chancery Lane, London, W.C.2.
Agents for the Applicants.

Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press (Leamington) Ltd.—1965. Published by The Patent Office, 25 Southampton Buildings, London, W.C.2, from which copies may be obtained.